

## DIFFUSION BONDING OF W/ODS STEEL USING PURE IRON INSERT FOR FUSION REACTOR APPLICATIONS

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### Abstract

The combination of tungsten (W) and oxide dispersion strengthened ferritic steel (ODS-FS) is considered to be adequate for the application to fusion diverter components. Joining of the two materials is hence essential to fabricate the required components. However, W cracks easily during the bonding process due to a large difference in the coefficients of thermal expansion (CTE) between these two materials. In this study, we attempted the reduction of the thermal stress using pure iron as an insert material for diffusion bonding of W and ODS-FS. The bonding strength of the bonded specimens was evaluated by four-point bend tests with a specially designed fixture. It was found that the bonding strength significantly depends on the grain structures such as grain size and grain orientation in W used for the W/ODS-FS joint. A much higher strength of the joint was obtained for the W with a relatively fine and equiaxed grain structure than for that with rolled or swaged grain structures.

### Introduction

Tungsten (W) is currently considered as a leading candidate for the plasma facing material (PFM) of ITER (International Thermonuclear Experimental Reactor) because of its high melting point (3695K), low hydrogen retention and superior resistance to spattering [1], although W is well known as a brittle metal. Oxide dispersion strengthened (ODS) steel has also been recognized as an advanced structural material for the fusion blanket systems with high thermal efficiency, since ODS steels exhibits superior mechanical properties at high temperatures to reduced activation ferritic steels [2]. Among the ODS steels, ODS ferritic steels (FS) are highly applicable to high-temperature structural components because of no transformation up to the liquid phase temperature and its low activation characteristics. A high performance Fe-15%Cr-2%W-0.2%Ti-0.35%Y<sub>2</sub>O<sub>3</sub>-ODS-FS was developed in Kyoto University: The oxide particles in the ODS-FS were reduced in size and increased in number density by simultaneous additions of small amount of Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> [3]. In order to reduce the temperature gradient between the PFM and supporting components, a higher operation temperature is required for the diverter systems. The combination of W and ODS-FS is expected for the fusion diverter components [4]. R&D in bonding these two materials is therefore a critical issue.

Problems to solve in this R&D of bonding are “relaxation of thermal stress caused by the large difference of CTE between W and ODS-FS. CTEs of ODS-FS and W are  $12.1 \times 10^{-6}/^{\circ}\text{C}$  and is  $4.5 \times 10^{-6}/^{\circ}\text{C}$  respectively and its difference may induce cracking of W. In our previous analysis of this thermal internal stress by FEM, the internal stress in W reached as high as 2 GPa [5]. In order to reduce the internal stresses, we employed an insert material between W and ODS-FS.

We at first selected a thin plate of nanostructured ODS V alloy that has the thermal conductivity and CTE between W and ODS-FS and reduced activation characteristics. However, diffusion bonded W/ODS-FS joints prepared with the V insert were very fragile and fractured in W in almost parallel direction to bonding interface during specimen cutting by electro-discharge, indicating that the use of the V insert does not reduce the internal stress sufficiently. In view of fairly high yield strength of the nanostructured ODS V alloy at ambient temperature where the thermal stress in the W/ODS-FS joints take a maximum, a ductile material with low yield strength at ambient temperature is expected to be effective. Therefore, in this study we have selected pure iron as an insert material: Pure iron is a low activation material and plastically more deformable in a wide range of temperatures after bonding than V alloys. Since the cracking for the diffusion bonded W/ODS-FS joints was found to always occur in the W close to the joined interface, the bonding strength of the joints may depend on the grain structures such as grain size and grain orientation in W used for the W/ODS-FS joint. The purpose of this study is to clarify the effects of W grain structures on the bonding strength of the W/ODS-FS joints and find W with the optimized grain structures.

### Experimental Procedure

#### 1. Specimen

The starting materials used in this study are commercially available W materials, ODS-FS (Fe-15%Cr-2%W-0.2Ti-0.35Y<sub>2</sub>O<sub>3</sub>) and pure iron as an insert material and listed in Table 1.

Table 1. Shape and Thickness or Diameter of Starting Materials Used in This Study.

Material (Purity, Supplier)	Shape	Thickness or Diameter (mm)
Pure Tungsten (99.95%, PLANSEE Co.)	Plate	1.0
		1.5
		2.0
		10.0
K-dope Tungsten (A.L.M.T. Corp.)		1.0
Pure Tungsten (99.95%, PLANSEE Co.)	Rod	φ20
Estan (99.95%, PLANSEE Co.)	Rectangular Block	25
Pure Iron (99.99%, PLANSEE Co.)	Plate	1.0
ODS-FS (Fe-15%Cr-2%W-0.2%Ti-0.35%Y <sub>2</sub> O <sub>3</sub> , KOBELCO Co.)	Rod	φ20

The pure W and K-doped W plates were hot forged and hot rolled and pure W rods were hot swaged. A block of Estan, which was fabricated via HIP (Hot Isostatic Press), exhibits an equiaxed grain structure. The ODS-FS was fabricated by mechanical alloying and hot extrusion and pure Fe by hot rolling. From the materials, specimens for joining or bending tests were machined by wire electro-discharge methods: The specimen dimensions for joining are 10 mm x 10 mm x 2.0 mm for the W and ODS-FS and 10 mm x 10 mm x 0.9 mm for pure Fe, and those for bending of the W materials are (1.0~2.0) mm x (1.0~2.0) mm x (7~13.2) mm. Figure 1 shows the designation of each specimen relative to the roll or extruded direction marked by the arrows. L and T stand for the longitudinal and transverse directions relative to the rolling or extruding directions, respectively. Specimen surfaces were mechanically polished with abrasive paper of #500 to 4000 and buff-polished with diamond paste with particle size of 1.0  $\mu\text{m}$ .

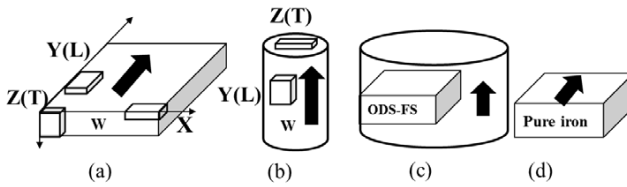


Figure 1. Designation of each specimen relative to the roll or extruded direction marked by the arrows. (a) W plate, (b) W rod, (c) ODS-FS rod and (d) pure Fe plate.

## 2. Diffusion Bonding and Bending Test

Diffusion bonding tests were carried out using the materials shown in Table 1. The insert material was placed between the tungsten and ODS-FS specimens so that uniaxial pressing yielded the joint of W and ODS-FS. Diffusion bonding was carried out at 1240°C for 1hr in a vacuum of  $8.0 \times 10^{-4}$  Pa with a hot press machine by applying 2% plastic deformation to the insert material. The bonding strengths of the diffusion bonded specimens ( $2.4\text{mm}^w \times 0.5\text{mm}^t \times 5.0\text{mm}^l$ ) were evaluated by miniature four-point bend tests with a specially designed fixture of the upper and lower spans of 0.8 and 3.0 mm, respectively at room temperature (Figure 2). Four-point bend testing has an advantage that the maximum flexural stress is constant between the upper span rods and effective in the evaluation of the joints. The flexural rate was  $1 \times 10^{-3}$  mm/s.

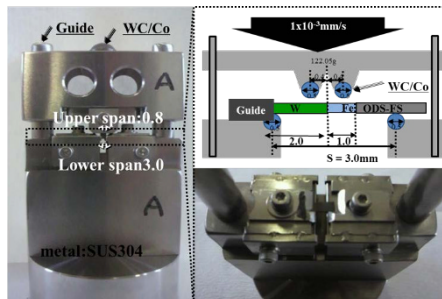


Figure 2. Miniature four-point bending test fixture.

Bend strengths of the W materials were measured in the as-received as well as the heat-treated state at 1240°C for 1hr that is the same as the bonding condition. Testing was conducted with three-point bending (span length: 7.0 mm or 13.2 mm) at room temperature. The fracture strengths of three point and four point bending were evaluated as the maximum fiber stress.

## Results and Discussion

Figure 3 shows the effects of thickness of the pure W plates and heat treatments at 1240°C for 1 hr on the fracture strength at room temperature. The strengths of the 1.0 mm, 1.5 mm and 10.0 mm thickness as-received W plates, Y(L), are 2440 MPa, 2150 MPa and 1230 MPa, that is, the strength significantly decreases with increasing W plate thickness. Since thin plates require a high degree of reduction ratio, the results obtained indicate that the strength of the as-received W increases with increasing rolling reduction ratio. This is because the plastic working such as hot rolling produces a high density of dislocations and grain refinement and generally increases the yield and fracture strengths.

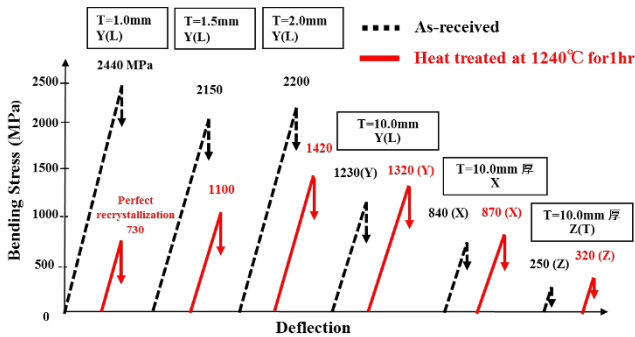


Figure 3. Three-point bending stress-strain curves for pure W plates ( $t = 1\sim 10$  mm) in the as-received and 1240°C -heat treated states.

On the other hand, the fracture strengths of the heat-treated W are 730 MPa, 1100 MPa and 1320 MPa, indicating that they increase with decreasing rolling reduction ratio. This is because a high degree of plastic working decreases the temperature at which recrystallization starts to occur. Recrystallization in W materials leads to the formation of new grain boundaries of random orientations (random grain boundaries) that are very high energy and very susceptible to cracking and these boundaries are prone to fracture as referred to as recrystallization embrittlement. The formation of random grain boundaries is attributed to strong covalence of atomic bonding in W and segregation and precipitation of gaseous interstitial elements of oxygen and nitrogen which are insoluble in the W [6]

For the 10 mm thickness specimen, the fracture strength increases by the heat treatments regardless of X, Y and Z directions, although the difference between the as-received and heat-treated conditions is small. This is probably due to the effect of recovery at 1240°C for 1 hr, because the degree of plastic working for the 10 mm thickness specimen is not high and may not cause recrystallization to start at 1240°C for 1 hr. However, the difference in strength between the X, Y and Z directions is remarkable. Especially, Z direction exhibits significantly low strength: The strengths of W for X and Z directions in the as-received states are 840 and 250 MPa, respectively, which are 68% and 20% of the strength for Y, respectively.

Figure 4 shows the effects of grain structures in W on three-point bending fracture strength at room temperature for three W materials with different fabrication processes. The strength of the K-dope W-Y(L) plate with 1.0 mm thickness is approximately the same as that of the 1.0 mm pure W-Y(L). The strength for X is 1460MPa, which is 60% of that for Y(L) and this vale of 60% is almost the same as that for that 10.0 mm pure W plate (68%). These results suggest that the plate thickness dependence of fracture strength for K-dope W is essentially the same as that of pure W plates.

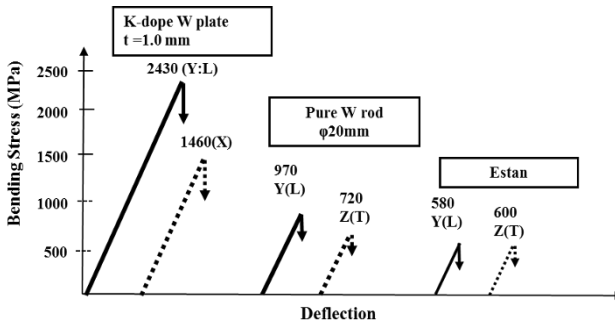


Figure 4. Three-point bending stress-strain curves for K-dope W plate, pure W bar and Estan that were fabricated with different fabrication processes and exhibit different grain sizes and grain orientations.

For the W rod, the fracture strengths for X and Y directions are much lower than those for K-dope W plate and a slight difference in strength is recognized between the X and Y directions. This is probably because the extrusion diameter of the rod is relatively large and thus the degree of plastic working is not high, as is almost the same as the case of the 10mm thickness plate in Figure 3. It should be noted that the fracture strength for Y(L) of Estan is the lowest among the as-received W materials investigated in this study and Estan does not exhibit any difference in strength between X and Y directions. This result for Estan is attributed to the aspect that Estan exhibits equiaxed grain structures produced by HIP and such grain boundaries are not strengthened and susceptible to cracking.

Figure 5 shows the effect of W grain structures on four-point bend fracture strength at room temperature for the W/ODS-FS joints with pure iron insert. Contrary to our previous results for W/ODS-FS joints with the V alloy insert that were very fragile and did not permit measurements of fracture strength, the present results for W/ODS-FS joints with pure iron insert clearly shows measurable fracture strengths. This means that the internal stresses produced in the joints were considerably reduced by using the pure iron insert due most likely to plastic deformation during cooling from 1240C to room temperature.

It is seen from Fig. 5 that the bonding strength for the K-Dope W plate is 112 MPa. Bonding with the T direction in which the grain orientation is parallel to the bonding surface yield extremely low strength: The pure W rod (T) was broken during specimen cutting before bending tests. On the other hand, bonding for L direction that the grain orientation is vertical against bonding surface yields much higher bonding strength than that for T direction: The bonding strength for the pure W bar (L) is 139 MPa. Compared to the two cases, Estan exhibits significantly high bonding strength, 265 MPa, although the fracture strength of Estan is the lowest among the tungsten materials used. This high bonding strength for Estan is attributable to

ease of the relaxation of the internal stress (thermal stress) produced in W in the W/ODS-FS joints when the W materials exhibit equiaxed grain structure. Therefore we can say that the W materials with equiaxed grain structure is suitable to enhancement of bonding strength for diffusion bonded W/ODS-FS joints with pure iron insert.

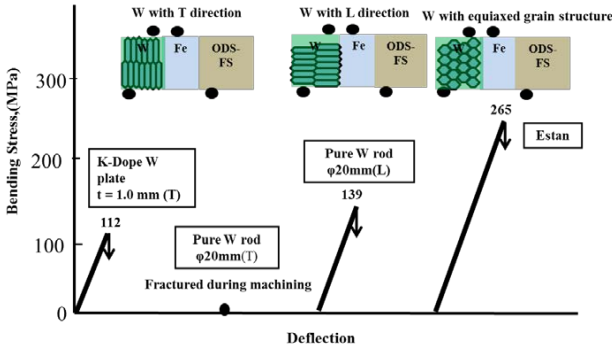


Figure 5. Effect of W grain structures on four-point bend fracture strength at room temperature for W/ODS-FS joints with pure iron insert.

In view of the weakening of grain boundaries in Estant, it is expected that the application of recently developed TFGR (Toughened, Fine Grained, Recrystallized) W-1.1%TiC to W/ODS-FS joints with pure iron insert leads to further increase in bonding strength of the joints, because TFGRW-1.1%TiC exhibits equiaxed, fine grain structures and random grain boundaries significantly strengthened by precipitation of TiC and segregation of the TiC constituents at the boundaries.

Further studies are needed to clarify the mechanism of the relaxation of the internal stress for W/ODS-FS joints with equiaxed grained W.

## Conclusions

The development of technology for diffusion bonding between W and ODS-FS requires relaxation of the thermal stress caused by the large difference of CTE between W and ODS-FS after bonding. In order to reduce the thermal stress, insertion of a ductile material with low yield strength between W and ODS-FS can be effective. Since the fracture of diffusion bonded W/ODS-FS joints was recognized to always occur at grain boundaries in W close to the bonding interface, the fracture strength of the joints may depend on the grain structures such as grain size and grain orientation in W. In this study, we have prepared W/ODS-FS joints by diffusion bonding at 1240 °C using pure iron insert and W materials with different grain structures and examined the effect of W grain structures on the bonding strength of the joints as well as on the fracture strength of W to find the optimized grain structures in W. The main results obtained are as follows:

1. The strengths of the as-received W plates significantly increase with decreasing plate thickness, indicating that the strength increases with increasing rolling reduction ratio.
2. The strengths of the W materials heat treated at 1240°C for 1 hr that is the same as the diffusion bonding condition significantly decrease with decreasing W plate thickness, indicating that they decrease with increasing rolling reduction ratio. This is because a high degree of

plastic working decreases the temperature at which recrystallization starts to occur and leads to recrystallization embrittlement.

3. For the 10 mm thickness plate, the difference in the strength between X, Y and Z directions is remarkable. Especially, Z direction exhibits significantly low strength.

4. The fracture strength of Estan is the lowest among the as-received W materials and Estan does not exhibit any differences in the strength between X and Y directions. This result is attributed to the aspect that Estan exhibits equiaxed grain structures produced by HIP and such grain boundaries are not strengthened and susceptible to cracking.

5. Contrary to our previous results for W/ODS-FS joints with the V alloy insert which did not permit measurements of fracture strength due to brittleness, the present results for W/ODS-FS joints with pure iron insert clearly show measurable fracture strengths.

6. The use of Estan as W leads to high bonding strength, 265MPa, although the fracture strength of Estan is the lowest among the W materials used. This high strength is attributable to ease of the equiaxial relaxation of the thermal stresses produced in W in the W/ODS-FS joints. Further studies are needed to clarify the mechanism of the relaxation of the internal stresses

7. W materials with equiaxed grain structure are suitable to the enhancement of bonding strength for diffusion bonded W/ODS-FS joints with pure iron insert.

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